

Ammonia Offgassing from SA9T

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NH₃ is a degradation product of SA9T, a solid-amine sorbent developed by Hamilton Sundstrand, that is continually emitted into the gas stream being conditioned by this sorbent. NH₃ offgassing rates were measured using FTIR spectroscopy using a packed bed at similar contact times as offgassing tests conducted at Hamilton Sundstrand and at the Ames Research Center. The bed was challenged with moist air at several flow rates and humidities and NH₃ concentration of the effluent was measured for several hours. The NH₃ offgassing rates in open-loop testing were calculated from the steady state outlet NH₃ concentration and flow rate. NH₃ offgassing rates from SA9T were found to be influenced by the contact time with the adsorbent (flow rate) and by the humidity of the inlet gas stream, which are consistent with previous studies. Closed-loop vacuum-swing adsorption cycling rates verified that NH₃ offgassing continues when a constant source of water vapor is present.

Nomenclature

CAMRAS	= Carbon Dioxide and Moisture Removal Amine Swing-bed
ECLSS	= Environmental Control and Life Support Systems
HS	= Hamilton Sundstrand
HSIR	= Human-Systems Integrations Requirements
PSA	= Pressure Swing Adsorption
SA9T	= Regenerable solid amine sorbent
RVCS	= Regenerable VOC Control System
SMAC	= Spacecraft Maximum Allowable Concentration
TC	= Trace Contaminant
VOC	= Volatile Organic Compound

I. Introduction

Spacecraft environmental control and life support systems (ECLSS) ensure suitable crew health and comfort by controlling human produced CO₂ (1 kg person⁻¹ day⁻¹) and water vapor (~2 kg person⁻¹ day⁻¹), and by removing trace contaminants (TCs) from cabin air^{1,2}. SA9T, a solid-amine sorbent developed by Hamilton Sundstrand (HS), is used in vacuum swing adsorption systems (e.g. the Carbon Dioxide and Moisture Removal Amine Swing-bed (CAMRAS)) to continuously remove and reject excess water vapor and carbon dioxide from the cabin to space vacuum, thereby eliminating the need for a condensing heat exchanger for cabin humidity control³. SA9T operates at ambient temperature and has the ability to remove select TCs during vacuum swing adsorption cycles^{3,4,5},

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whereby TCs are adsorbed onto the sorbent during the loading ½-cycle and they are desorbed to vacuum during the desorption ½-cycle.

NH₃ is a degradation byproduct of SA9T in the presence of water that is continually emitted into the gas stream being conditioned by this sorbent³. NH₃ is listed in the Constellation Program Human-Systems Integrations Requirements (HSIR) Document as a gaseous pollutant that must be controlled in habitable volumes to below to less than 50% of its Spacecraft Maximum Allowable Concentration (SMAC) level of 3.5 mg m⁻³ (5 ppm). Therefore, the offgassing properties of SA9T have been tested by the White Sands Test Facility (WSTF) in 2003 and by HS in 2005 (Papale, 2007). The WSTF test followed NASA-STD-6001 Test #7 and concluded that SA9T offgassed 370 mg NH₃/g (5.1 mg NH₃ hr⁻¹ kg⁻¹) after heating SA9T at 49°C for 72 hr. Testing by HS using Draeger tubes found that 3 mg NH₃ hr⁻¹ kg⁻¹ were generated at nominal conditions in a closed sample volume. In an open flow system using HCl traps to capture NH₃, HS found that 1.2 mg NH₃ hr⁻¹ kg⁻¹ were generated at the 0.25 s contact time used in CAMRAS. Recently, NH₃ emission rates ranging from 0.9 to 2.9 mg NH₃ hr⁻¹ kg⁻¹ were measured at the Ames Research Center (ARC) in 2009⁷. The tests at ARC also identified that higher emission rates of NH₃ were observed as the humidity of the air stream increased. This study presents systematic measurements of NH₃ offgassing rates during dynamic conditions found within air revitalization systems utilizing SA9T for humidity and CO₂ control.

II. Materials and Methods

A. Regenerable VOC Control System (RVCS)

The NH₃ offgassing rates of SA9T were measured at NASA's Kennedy Space Center (KSC) in the Regenerable VOC Control System (RVCS), a sub-scale tesbed⁸. The RVCS consists of the following subsystems: CO₂ mixer, humidifier, gas generator, instrumented adsorbent bed with dual side vacuum regeneration, automated sampling of inlet and outlet gas stream relative humidities, VOC composition, and CO₂ concentrations. The RVCS is capable of measuring VOC, CO₂ and water vapor breakthrough curves (static tests) and exposing the bed to programmed vacuum swing adsorption cycles (dynamic tests)^{8,9,10}. A Fourier Transform Infra-Red (FTIR) spectrometer was added to the RVCS to allow the measurement of NH₃ to a detection limit of 0.13 ppm. The Gasmeter Dx-4030 (Gasmeter Technologies, Inc., Bellingham, WA) FTIR spectrometer has a Rhodium-Gold coated sample cell (0.48 L), a spectral resolution of 8 cm⁻¹ and scans between 900-4200 cm⁻¹. It has a built-in sample gas pump and operates using a flow rate up to 2 l/min with a multi-pass, fixed path length of 9.8 m.

B. Test Conditions

SA9T beads (~7g) were held vertically in a 1 inch diameter bed, within a 2.54 cm long metal wire mesh frame⁹. The aluminum foam used for thermal linking of SA9T in CAMRAS was not used. The bed was insulated and instrumented with an inner bed temperature sensor. Essentially the RVCS measures adsorption through a 1 inch cylinder that is 1 inch long. This geometry approximates the 1 inch cross-section of an aluminum foam segment of a single adsorbent layer in CAMRAS. The bed was instrumented with inner and outer bed temperature sensors, inlet and a outlet pressure and relative humidity sensors. The flow rate (2.9 L min⁻¹) for the tests was chosen to match the contact time (0.25 s) of previous tests conducted by HS and ARC. The tests were conducted using an airstream at 23°C and varying relative humidities. The CO₂ concentration used was 6000 ppm. The SA9T bed was regenerated after each test by desorption in a vacuum at <0.2 torr for >3 hours, which proved to be sufficient for removing all CO₂, H₂O vapor, and most VOCs from the sorbent.

C. Gas Analysis - FTIR

NH₃ offgassed from SA9T was measured using an FTIR spectrometer. FTIR spectroscopy is based on the principle that individual gases have distinct infrared absorption features and a VOC is quantified by its molecular absorption of infra-red radiation at specific wavelengths. This enables the simultaneous measurement of several gases with one instrument since every IR spectrum contains the information of all IR radiation absorbing gases between a radiation source and a detector. NH₃ has a strong spectrum relatively clear of other absorption except for isolated H₂O lines.

The Gasmeter FTIR accurately measures low VOC concentrations as long as the specific IR adsorption lines for the target gas does not suffer from significant interferences from the IR adsorption spectra of water vapor and CO₂, the predominant IR absorbers, or from other VOCs. The FTIR assumes a linear response to concentration, and the slope and intercept of the line have been determined from factory calibration libraries. The calibration developed from the factory library was checked against an NH₃ gas standard. A calibration curve (3.4, 6.3, 5.6, and 9.8 ppm) was created by diluting a 9.8 ppm NH₃ gas standard using N₂ and two mass flow controllers. Although the lowest

NH₃ concentration that could be attained with this setup was 3.4 ppm, it was sufficient to verify that the FTIR response is linear at lower concentrations. The 9.8 ppm NH₃ gas standard was read as 9.4 ppm by the FTIR.

D. Artifacts – wall materials

NH₃ Ammonia measurements can suffer from inlet wall interactions along sampling tubes due to the highly reactive nature of NH₃. These are important because they attenuate high frequency fluctuations in NH₃ concentration¹¹. An analysis of sample tube materials found that the attenuation of high frequency NH₃ concentration fluctuations was most likely due to adsorption and desorption to the sample tube walls¹². These tests suggested that polyethylene was the best material when compared to PTFE, or heated silicoteel. Stainless steel was ~4 times more adsorptive than polyethylene. Heating was found to hinder NH₃ adsorption to walls but did not seem to affect desorption. The lines connecting the FTIR to the SA9T bed were PTFE because although polyethylene adsorbs less NH₃, it adsorbs more water than PTFE. NH₃ is undoubtedly adsorbed onto the sampling lines and the walls of the FTIR, however, the amount of NH₃ coated onto the walls and its influence on NH₃ concentration measurements is minimized by keeping the lines between the FTIR and the bed short and because the NH₃ concentrations to be measured are not fluctuating. During NH₃ calibration, the signal took ~60-80s to reach its final concentration.

E. Sampling Geometries

1.1. Closed-loop Test

Offgassed compounds accumulate within the SA9T bed when there is no flow and a closed loop sampling geometry (Fig. 1) was used to measure the VOCs trapped in the interstitial headspaces between the SA9T beads. The SA9T bed was placed in series with the FTIR. The bed was sealed and VOCs were allowed to offgas from the beads for a suitable amount of time to reach steady state (e.g. overnight). The internal pump of the FTIR was used to recirculate air through the bed, the tubing, and the 0.4 L cell of the FTIR at 0.4 Lpm. The interstitial NH₃ concentration was measured every 20 seconds, while the FTIR pump was turned on (Fig. 2). Interstitial ammonia from 6.5g of SA9T increased the NH₃ concentration in the FTIR cell up to 10 ppm in ~100 s. From this observation, the interstitial NH₃ contained in SA9T is estimated to be 0.56 mg/kg.

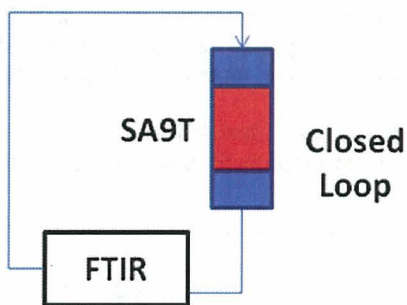


Figure 1. Closed Loop test

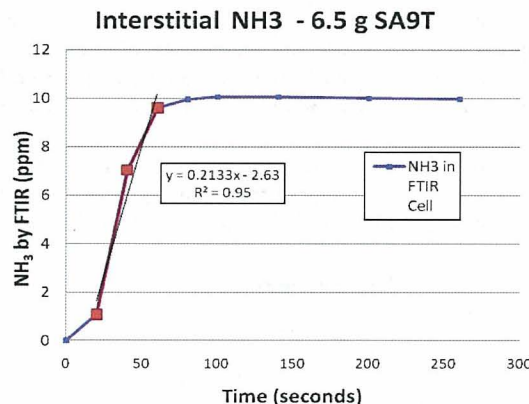


Figure 2. NH₃ held within interstitial spaces of SA9T accumulates in closed-loop configuration.

1.2. Open-loop Test

The rate of NH₃ offgassing from SA9T was measured using an open loop configuration (Fig. 3). The gas generator of the RVCS testbed was used for providing moist air at different relative humidities and flow rates (Monje et al, 2010a). For these tests, the SA9T was first vacuum desorbed for ~2 hours to clean the SA9T. Then, a gas stream at a constant relative humidity was mixed and fed to the SA9T bed. The flow rates were changed to vary the superficial velocity and the contact times. The steady state outlet NH₃ concentrations (measured after 8 hours) at each flow rate were used to calculate the NH₃ offgassing rate. The offgassing rate (expressed in mgNH₃ kg⁻¹hr⁻¹) was calculated from the steady state NH₃ concentration and the flow rate of the outlet gas stream.

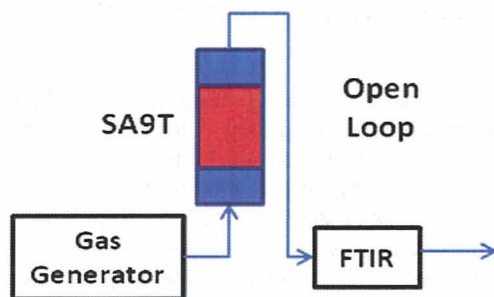


Figure 3. Open Loop test

When wet SA9T is allowed to sit stagnant for several days, NH_3 , acetonitrile, and acrylonitrile accumulate within the interstitial spaces. Thus, when flow (0.4 slpm - 1.9 sec contact time) was allowed to carry these gases from the bed, large peaks of NH_3 (~90 ppm) and acetonitrile (~12 ppm) were observed in the outlet gas (Figure 4). The acrylonitrile was washed out after 0.3 hrs, but outlet NH_3 concentration remained high at ~3 ppm after 1 hr.

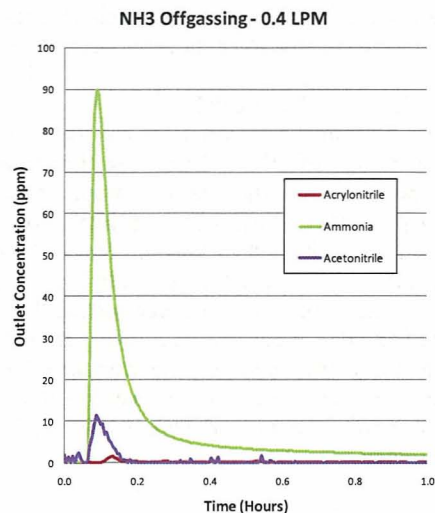


Figure 4. Acrylonitrile NH_3 , Acetonitrile, and offgassing from Stagnant SA9T.

1.3. Outlet NH_3 Concentration

The NH_3 concentration in the outlet air stream of a bed containing 6.5 g of SA9T was measured at a flow rate of 0.4 slpm (1.9 sec contact time). The NH_3 concentration took ~80 to 100s to equilibrate following a change in humidity (Fig. 5). Outlet NH_3 concentration increased as the humidity of the inlet gas stream was increased. The NH_3 concentration of the outlet air reached 1.8 ppm at 63% RH, 0.8 ppm at 32% RH, 0.4 ppm at 15% RH, and 0.2 ppm at 5% RH. The outlet NH_3 concentration takes several hours (4-5 h) to reach a steady state at this flow rate. This implies that short term measurements of outlet NH_3 concentration will overestimate the steady state NH_3 offgassing rate. The length of time for the NH_3 offgas rate from SA9T to reach steady state depends on the humidity of the inlet air. It also depends on the flow rate because higher flow rates dilute the amount of NH_3 given off by SA9T.

The outlet NH_3 concentration decreased rapidly when the flow rate was increased from 0.4 slpm (1.9 s contact time) to 1.6 slpm (0.5 s contact time) (Fig. 6). After 12 min, the outlet NH_3 concentration drops to 0.43 ppm at 60% RH, 0.21 ppm at 30% RH, and 0.15 ppm at 15% RH.

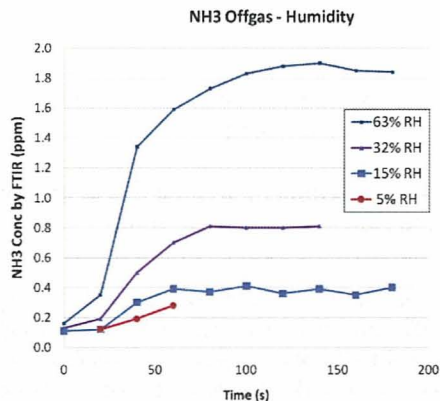


Figure 5. NH_3 offgassing rate at 0.4 slpm increases as the humidity of the inlet increases.

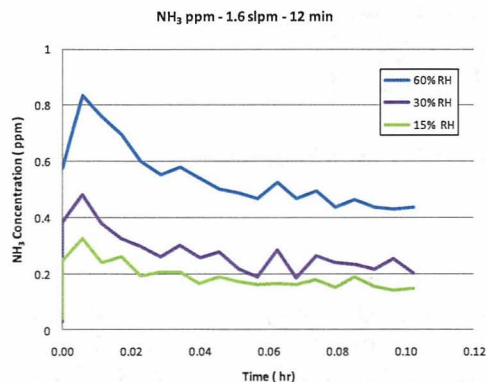


Figure 6. NH_3 offgassing rate at 1.6 slpm decreased rapidly reaching steady state after 2 hours.

1.4. NH₃ Offgas rate – Contact Time

The NH₃ offgassing rates at 60% and at 30% RH measured after 12 minutes were compared at 3 flow rates (0.4, 1.6, and 3 slpm) corresponding to (1.9, 0.5, and 0.25 s) contact times (Fig. 7). The 12-minute NH₃ offgassing rate at 60% RH was found to be strongly affected by contact time. NH₃ offgassing rates increase initially as contact time is reduced to 0.5 s, but are further reduced as contact time decreases to 0.25s. A similar relation was observed at 30% RH, but NH₃ offgassing rate plateaus form moist SA9T plateaus near 2.2 mg NH₃ kg⁻¹hr⁻¹ at 0.25s.

1.5. NH₃ Offgas rate – Steady State

At a contact time of 0.25 s (2.9 slpm), the NH₃ offgas rate from SA9T takes ~2-3 hours to reach steady state. The NH₃ offgas rate, calculated from the NH₃ concentration rises initially and declines steadily over 2 hours, until a steady state is reached. The initial rise in NH₃ offgas rate was higher as the inlet humidity was increased. At 60% RH and 0.25s contact time, the offgas rate decreased from 11 to 2.5 mg NH₃ kg⁻¹hr⁻¹ over 2 hours (Fig 8).

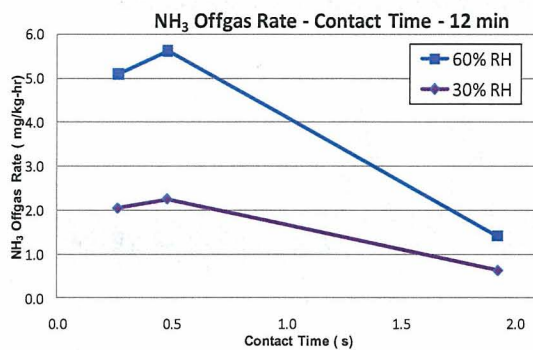


Figure 7. Effect of contact time on NH₃ offgassing rate.

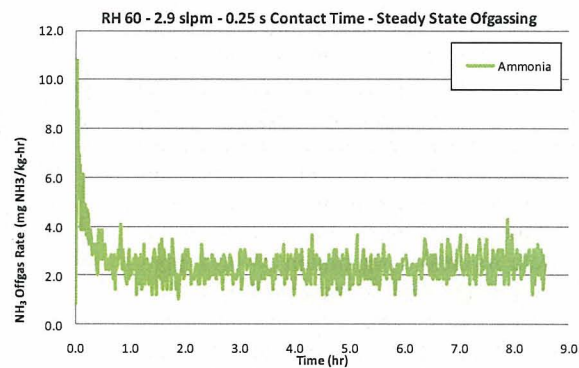


Figure 8. Steady state NH₃ offgassing rate at contact time found in CAMRAS.

1.6. NH₃ Offgas rate – Gas Stream Humidity

The NH₃ offgassing rate from SA9T was strongly dependent on humidity, which confirms the observations at ARC⁷ (Fig. 9; red circles, ARC). The ARC data shows greater offgassing rate at low humidity compared to KSC (blue diamonds) probably due to errors from crosstalk between NH₃ and water vapor in the Picarro spectrometer. Figure 7 shows that NH₃ offgassing rates ranging from 2-3 mg NH₃ kg⁻¹ hr⁻¹ can be expected at 60% inlet relative humidity. The range of NH₃ offgassing rates reported by HS (1.4-5.1 mg NH₃ kg⁻¹ hr⁻¹) (square; arbitrarily placed at 45% relative humidity) brackets the range of values observed at ARC and KSC.

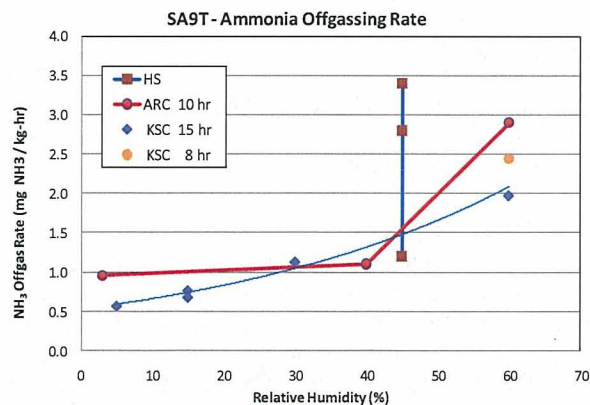


Figure 9. NH₃ Offgas rates from SA9T as a function of inlet relative humidity.

2. Dynamic Tests

2.1. Conditions

The dynamic test consisted of exposing an SA9T sample to 40-minute and 6-minute PSA cycles. During each PSA cycle the bed alternates from the open-loop configuration to the recycle configuration¹⁰ (Fig.3 to Fig. 10). In open-loop a humid air stream passes over the bed and NH_3 offgassing from the bed is measured by the FTIR. During the recycle configuration, the SA9T bed is regenerated by vacuum desorption (<0.3 torr). CO_2 (0.6%) was also provided in the gas stream to simulate nominal CAMRAS conditions. The RVCS plumbing was modified in order to monitor NH_3 concentration with FTIR.

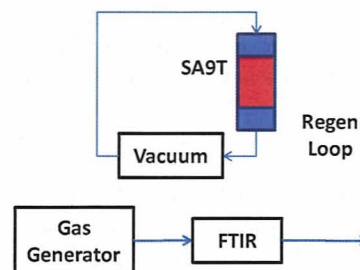


Figure 10. Dynamic test

2.2. PSA Cycles

SA9T NH_3 offgassing was measured during 40-min and 6-min vacuum swing cycles using 60% RH air at a contact time of 0.25 s (Fig. 11). First, the SA9T bed was vacuum desorbed for 2 hours. Then, the bed was exposed to three 40-min vacuum swing cycles until the response was stable. Outlet NH_3 concentration during the 20-min loading half-cycle reached a maximum of 0.31 ppm, and an average NH_3 concentration of 0.15 ppm. This corresponds to maximum and average offgassing rates of 6.3 and 3.05 $\text{mg NH}_3 \text{ kg}^{-1} \text{ hr}^{-1}$, which are 2.5 and 1.2 times greater than the steady state offgassing rate at (2.5 $\text{mg NH}_3 \text{ kg}^{-1} \text{ hr}^{-1}$). In contrast, the outlet NH_3 concentration during the 3-min half-cycle only reaches a maximum concentration of 0.15 ppm, and an average of 0.09 ppm. Thus, at high flow rates the maximum and average offgassing rates of NH_3 are 3.0 and 1.75 $\text{mg NH}_3 \text{ kg}^{-1} \text{ hr}^{-1}$.

Since CAMRAS has two alternating beds to maintain a constant water vapor and CO_2 removal, then the constant NH_3 offgassing rate should equal the rate determined during the loading half-cycle of the vacuum swing cycle. Therefore, the NH_3 production rate at 6-min cycles is expected to be 1.8 $\text{mg NH}_3 \text{ kg}^{-1} \text{ hr}^{-1}$. This dynamic NH_3 offgassing rate is 28% lower than the 2.5 $\text{mg NH}_3 \text{ kg}^{-1} \text{ hr}^{-1}$ offgassing rate predicted from steady state measurements at 60% RH.

2.3. Dynamic Closed Loop Offgassing Tests

A 20L chamber was connected in series with the 0.4L cell of the FTIR and the SA9T bed (Fig. 12). The FTIR pump was used to recirculate the air in this closed loop at 2.9 slpm (contact time of 0.28 s). The leak rate of the chamber was found to be 2%/hr using CO_2 . The SA9T bed would deplete any CO_2 or water vapor in the chamber, and compounds offgassed from SA9T would accumulate in the chamber. The SA9T bed was regenerated using programmable PSA cycles. The chamber was used to follow what happens to gases in contact with a cycling SA9T bed in a closed-loop. It is expected that gases could be removed by both the chamber leak rate and by loss of ullage air during each vacuum desorption cycle. The SA9T bed was vacuum desorbed for 2 hours before starting the tests

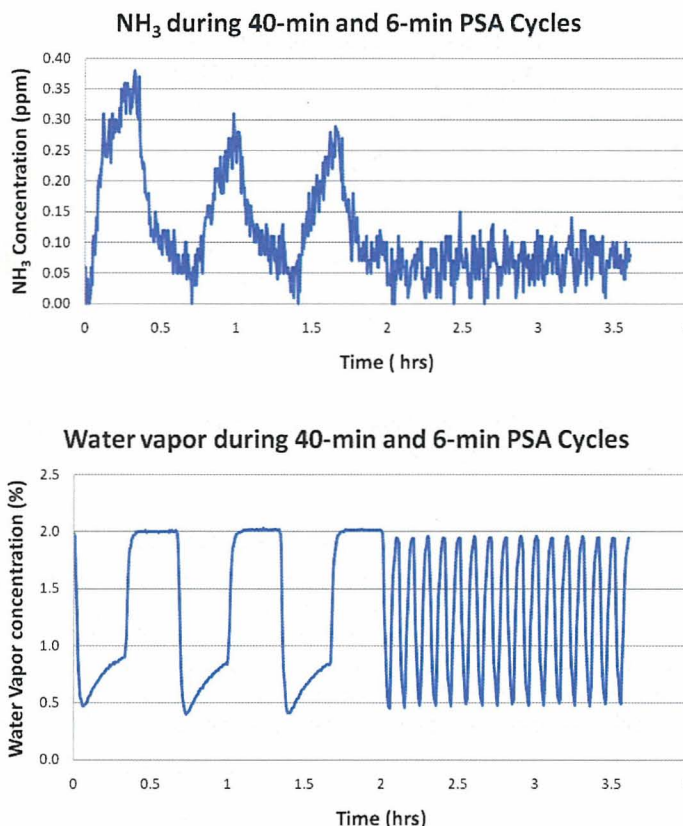


Figure 11. Outlet NH_3 and water vapor concentration during vacuum swing cycling.

in order to have a fresh bed. Water vapor and NH_3 concentrations of the recirculated air were measured every 20 seconds.

NH_3 concentration increased rapidly when the water concentration in the chamber was increased in the 1st hour of the experiment. NH_3 concentration then declined exponentially to a steady state concentration ~ 0.2 ppm after 16 hr. The water vapor in the chamber also declined reaching steady state $\sim 0.2\%$.

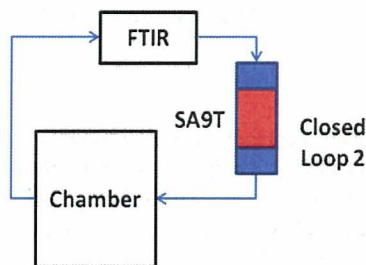


Figure 12. Dynamic Closed Loop test

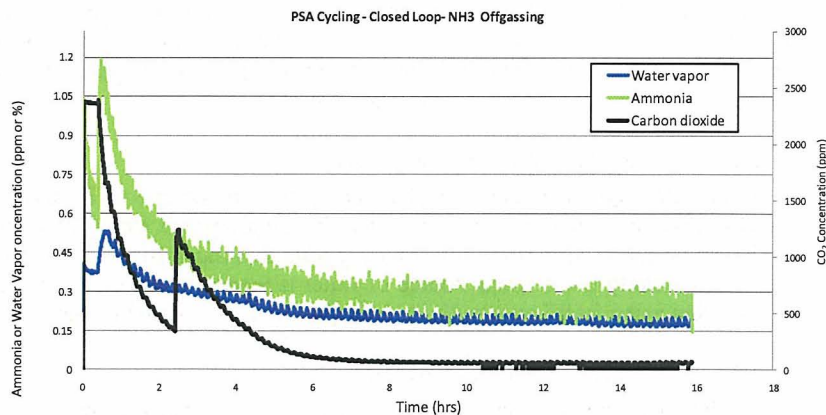


Figure 13. NH_3 Concentration in a closed 20L chamber undergoing 10 min vacuum swing cycles decreased steadily and reached steady state after 8 hrs

The NH_3 concentration in the closed system (Fig. 13) decreased more slowly than in the open loop system (data for generating Fig. 9). It must be noted that there was not a continuous input of humidity into the chamber and therefore NH_3 decreased as the chamber was dried by the SA9T bed undergoing 10 minute vacuum swing cycles. A continuous emission of NH_3 is expected at room temperature since only trace amounts of water vapor are needed to sustain the degradation rate of SA9T (Papale, 2008). CO_2 concentration was 2500 ppm initially and began decreasing each vacuum swing cycle. CO_2 was added after 2 hours and the SA9T bed removed it over the next 12 hours.

III. Results and Discussion

2.4. Overview

NH_3 is a degradation product of SA9T that is continually emitted into the gas stream (Papale, 2008)³. In order to determine the magnitude of NH_3 emitted from SA9T, NH_3 offgassing rates were measured using FTIR spectroscopy. These measurements were conducted at the Kennedy Space Center using a bed containing 7g of SA9T at similar contact times as offgassing tests conducted at Hamilton Sundstrand and at the Ames Research Center. The geometry of the bed in this study was that of a 1 inch long by 1 inch in diameter bed of SA9T. Furthermore, SA9T was not held in thermal-cycling aluminum foam, like in CAMRAS. The bed was challenged with moist air at several flow rates and humidities and NH_3 concentration of the effluent was measured for several hours. The NH_3 offgassing rates were calculated from the steady state outlet NH_3 concentration and flow rate.

2.5. Results

NH_3 was offgassed in open-loop tests at fast flow rates for several hours, after which the offgas rate equilibrated. NH_3 offgassing rates were overestimated when measured after short times of exposure to flow rate because the SA9T emission was not at equilibrium due to residual NH_3 within interstitial spaces. NH_3 offgassing rates from SA9T were found to be influenced by the contact time with the adsorbent (flow rate) and by the humidity of the inlet gas stream (Fig. 7). These observations are consistent with those of HS³, who found that despite purging the beds to vacuum for 5 minutes and air flowing through the system for several hours, the rate of NH_3 permeation from the sorbent was relatively slow, eventually stabilizing to the steady values observed between 3 to 6 operating

hours. The response of SA9T NH_3 offgas rates to relative humidity observed by ARC was confirmed in open-loop testing.

HS³ suggested that because the degradation reaction is relatively slow at room temperature, only trace amounts of water vapor are needed to sustain the NH_3 offgassing. Closed-loop PSA cycling rates verified that NH_3 offgassing continues at low moisture contents (Fig 9; 0.2 % water vapor). These tests suggest that NH_3 concentrations may be high in small closed volumes if a constant source of water vapor is present (e.g. 0.3-1 ppm NH_3 in a 20 L chamber).

Measurements of acetonitrile offgassing were attempted using the current gas analysis system, however, interferences from pentane in the gas streams have to be resolved and a suitable analytical procedure developed. The source of the pentane must be identified, but it is probably a contaminant present in the industrial grade N_2 and CO_2 gases used for mixing the simulated gas streams.

2.6. Procedural Recommendations

The results suggest that air revitalization systems utilizing SA9T must be vacuum regenerated and vented for several hours after sitting stagnant before use. Such a procedure would avoid high rates of NH_3 offgassing during the initial start-up phase. Closed-loop testing suggests that much higher NH_3 concentrations can be expected in small volumes, especially if a constant source of water is present. Without a constant source of water, the cyclic vacuum swing system using SA9T eventually dries the bed and NH_3 emission rate becomes stable (Fig. 13).

2.7. System NH_3 Generation Rate

The NH_3 emission rate data obtained is useful for predicting the daily NH_3 production rate by a CAMRAS-like system. The CAMRAS Unit #3 contains ~112 gm of SA9T per column, 5 columns per layer, 3 layers per bed, and 2 beds per unit. Thus, the total mass of SA9T is 3360 g. Since only 1 bed is always in contact with the air, then 1.7 kg of SA9T are offgassing NH_3 into the cabin. The CAMRAS system NH_3 production rates were calculated using measured NH_3 offgassing rates (Table 1). This comparison shows that PSA cycling helps reduce the amount of system NH_3 production, probably because the

Table 1. Comparison of SA9T NH_3 Offgassing and Estimated CAMRAS Daily System NH_3 Production Rates.

Parameter	WSTF 2003	HS 2008	ARC 2007	KSC 2010	KSC 2010	KSC 2010	
NH_3 Offgas Rate	5.14	1.4	2.9	1.1	2.5	1.8	$\text{mg kg}^{-1} \text{ hr}^{-1}$
Sample Mass	21	1700	1	6.5	6.5	6.5	g
Temperature	49	24	24	23	23	23	$^{\circ}\text{C}$
Relative Humidity	na	na	60	30	60	60	%
Contact time	na	0.26	0.27	0.27	0.27	0.27	s
Run Time	na	2	2	10	10	<0.2	hours
PSA Cycling	no	no	no	no	no	yes	
System NH_3 Production	209	57	118	45	102	73	mg day^{-1}

IV. Conclusions

The adsorptive capacities for SA9T were measured systematically using gas streams containing single-compound and mixtures of VOCs. The presence of VOCs affected the CO_2 and water vapor adsorptive capacities of SA9T. In contrast to previous studies, the presence of water vapor did not increase CO_2 adsorptive capacity of SA9T and the presence of CO_2 interacted with the amount of water vapor adsorbed by SA9T. This work suggests that while

SA9T provides some VOC removal that may contribute to smaller trace contaminant control equipment, it does not, under a conservative design strategy, eliminate the need for a dedicated trace contaminant control function/unit operation and equipment.

Acknowledgments

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